

PROJECT ADMINISTRATION DATA SHEET

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ADMINISTRATIVE DATA

1) Sponsor Technical Contact:

Dr. Gary Kekelis

Code 4130

Naval Coastal Systems Center

Panama City, FL 32407-5000

(904) 234-4472

OCA Contact

R. Dennis Farmer x-4820

2) Sponsor Admin/Contractual Matters:

Mr. Thomas A. Bryant

Office of Naval Research

Resident Representative

206 O'Keefe Building

Georgia Institute of Technology

Atlanta, GA 30332-0490 (404)881-4374

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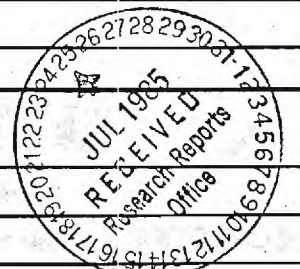
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Includes Subproject No.(s)N/A

Project Director(s)D. T. ParisGTRC X GTRXX

SponsorNaval Coastal Systems Center, Panama City, FL 32407

TitleInvestigations on Super Conducting Wire and Thin Film

Effective Completion Date: *8/19/86 (Performance) *8/19/86 (Reports)

Grant/Contract Closeout Actions Remaining: *See letter from Donald Calder to Faith Gleason dtd. 12/5/86

- ☐ None
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INTERIM REPORT ON CONTRACT

N61331-85-D-0025

W. G. MOULTON

One major emphasis of the work carried out under this contract has been to carry out further development of a system to detect and measure flux motion in very small magnetic fields under the influence of thermal gradients. The final results are relate these measurements to the noise generated in gradiometers due to flux motion generated by thermal gradients, and to develop criteria for choice of materials to minimize such noise. Descriptions of the first system designed to make such measurements is contained in previous technical reports submitted to NCSC and NRL. A major problem in this first system was the heating of the pickup loop when a thermal gradient was applied across the sample in which flux motion was to be measured. In fact, we were able to show that all the data taken with this system involved sufficient flux motion in the pickup loop to prevent any quantitative data on the sample to be taken. The system was redesigned and major modifcations were made in the machine shop to rectify this problem. The key to the solution of this problem was to provide a much stronger coupling to the He bath by modifying the post on which the pickup loop is mounted so that He can surround the line to the pickup loop and be in contact with the bottom of the post. This was accomplished by drilling a hole nearly the entire length of the new post and using a Stycast seal at the bottom of the post to provide the

transition to the vacuum environment of the chamber in which the measurements are made. An additional modification was made in the posts connecting the sample to the heaters and the liquid He bath so that the thermal coupling of the sample to the bath could be easily adjusted. A schematic of the new system is shown in Figure 1.

This system works very well in holding the temperature of the loop constant. Application of a 20K gradient across the film now results in only a 0.02K rise in temperature of the loop. With a copper film in place no signal was observed from the SQUID and loop after cooling in a 10G field (supplied, by a superconducting coil below the sample which is switched off after the sample has been cooled in the field), and a 20K thermal gradient is applied across the copper film.

In subsequent measurements with a Nb film ($T_c=9.2K$) in place, the film and loop were cooled in the ambient magnetic field in the can ($\sim 10^{-2}$ gauss), the film heated to 20K (above T_c) and then cooled in a magnetic field. The field was then removed and thermal gradients applied to the film. No signal was observed far below the transition temperature unless the magnetic field was at least 8 gauss and a thermal gradient of 4K applied across the film. This signal was reversible and based on calibrations described later, corresponded to a flux change of about $15\Phi_0$. When the film was heated above the transition temperature a large signal was observed, indicating flux was

being trapped in the film.

At the time these measurements were made we did not have an experimental calibration of the system. However, it did not seem reasonable that such large fields and temperature gradients should be required to observe flux motion, if indeed flux was being trapped in the film, as indicated by the signal when the film was heated above the transition temperature.

From a calculation of the mismatch between the pickup loop and the SQUID and the noise observed when the SQUID was shorted, it should have been possible to detect a flux change of $0.1 \Phi_0$ in the pickup loop. However, when the pickup loop was connected to the SQUID, the noise was found to increase by a factor of ten to twenty rather than about a factor of two as expected. The magnetic field of the superconducting solenoid as a function of current at the pickup loop was measured with a flux gate magnetometer and calculated from the geometry, giving a calibration of $5.75 \mu\text{A}/\Phi_0$ at the loop. Subsequent measurements showed that a current of $29 \mu\text{A}$ was required to produce a signal at the SQUID corresponding to $5\Phi_0$.

The origin of this excess noise was initially not clear and improvements had to be made to provide the information required. It was clear that the origin was probably not r.f. pickup since previous experience showed that when this is a problem, placement and connections of cables to heaters, thermometers, etc. change the noise level dramatically. The connection of the pickup loop

at the SQUID and the connections of the loop to the SQUID have been redone. Also, the superconducting shielding of pickup loop and the measurement chamber have been improved. These changes reduced the excess noise due to the pickup loop to that expected, namely a factor of 2. The sensitivity at the loop is now $0.2\Phi_0$ and measurements on films will begin immediately.

Another important aspect of this work has been the development and evaluation of ion implanted superconducting films as potential new shielding materials. An extensive study of the δ -NbN system produced by ion implantation was carried out under previous NCSC and NRL contracts and is described in reports on the contracts. While this system showed promise it is somewhat limited by the relatively low T_c of 10.5 K that it was possible to achieve. Some recent reports on laser annealed MoC produced by ion-implantation show that T_c 's of 16K may be achieved. On this basis we have turned our attention to this material. Considerable development work has gone into the preparation of Mo films in our vacuum system for this work. Good quality films have been produced, although the optimum conditions are somewhat different than for Nb. For example, considerably higher evaporation rates are required, 150 Å/sec. for Mo as compared to 60 Å/sec. for Nb. Residual resistance ratios are about 5. Implantation and T_c measurements of these films will begin shortly.

Another development which will greatly enhance the Florida

State effort is the addition of Dr. Louis Testardi to the faculty. Dr. Testardi is a well known materials scientist and is very interested in participating in this work. He is ordering the components of an ultra-high vacuum thin film system which will add greatly to the capability in this area.

Deliverables which have been delivered during this period are two new sets of vacuum feed throughs for the new measuring system previously constructed in the Physics department shops and delivered to NCSC. These new components were constructed in the Physics department machine shops. In addition, Nb foil rolled from a nearly single crystal Nb slug prepared at Florida State has been delivered to Sperry for evaluation and incorporation in a new design by them.

I have participated in two consultations with NCSC and NRL personnel at NCSC. At one of these consultations, Sperry personnel were present and future plans were made for the next stages for development of this program. A summary of this meeting has been distributed by Dr. Stuart Wolf of NRL.

The following professional personnel have participated in this research:

Dr. W. G. Moulton, Principal Investigator

Michael Goldstein, Graduate Student

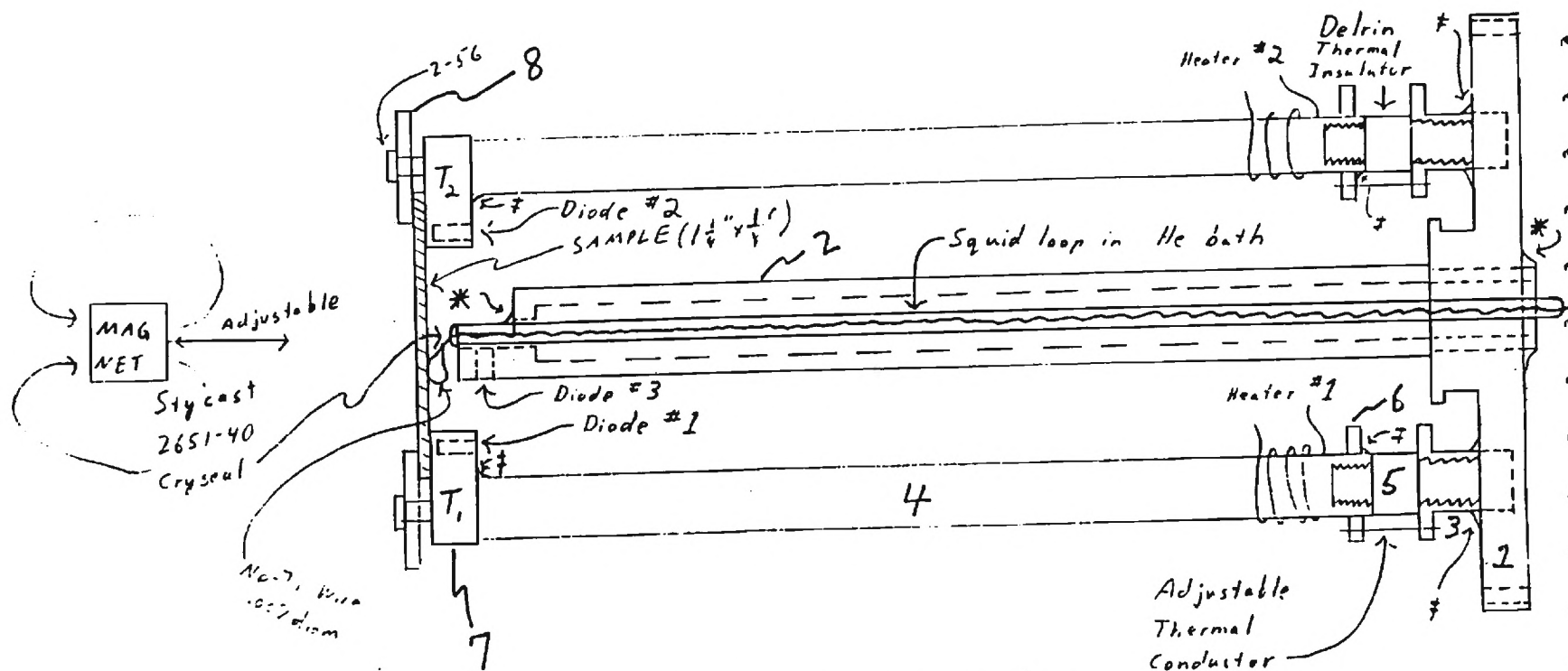
John Graham, Senior Graduate Student

Spencer Trimble, Engineer III

Laurence Peirce, Staff Physicist

FIGURE 1

SQUID PROBE ASSEMBLY



**FINAL REPORT ON SUBCONTRACT E-21-J-02-S1
UNDER NCSC CONTRACT NO. N61331-85-D-0025**

**William G. Moulton
Florida State University**

June 25, 1986

INTRODUCTION

This work is part of a multifaceted program involving NCSC, NRL, and Florida State University, to improve the performance of SQUID gradiometers and magnetometers operating in the field. It has been established by this team that one component of the system noise is associated with the motion of flux trapped in the shields, loops, or SQUIDS. This also may be a source of the $1/f$ noise. It was felt that it was likely that the motion of the flux was due to temperature changes or temperature gradients due to the sloshing of the He bath in field situations. The purpose of this part of the work is to study existing, and produce new, materials with different flux pinning characteristics, and study the behavior of the trapped flux motion with known temperature changes and gradients to help choose the best materials for shields and components of a system. Since no such study at very low magnetic fields has been made previously, it is also important to examine and understand the flux flow behavior in order to help optimize a system.

During this phase of the contract we have concentrated on measurements of the motion of flux trapped by very small (10^{-5} to 2.25 mT) magnetic fields in thin film materials. A number of problems were encountered with defective thermometers and vacuum leaks, which have now been solved. As stated in earlier reports on this aspect of the work, these measurements push the frontier

of the state of the art. In spite of these difficulties, considerable data have been taken which show very interesting behavior which should be of considerable significance in developing better solutions to the magnetic shielding problem. Since the results of the measurements were unexpected, it was felt that it was important to carry out more detailed studies on a few different kinds of films than to do only cursory studies of a larger number of different kinds of films.

LEAD DATA

The data were taken on 2000Å thick Pb films with a T_c of 7.4K. The films were prepared by evaporation at 10^{-7} T using resistive heating. The data were taken by cooling the films to 4.2K in the SQUID system described in previous reports to NCSC and NRL on this work. The cool down occurred in the ambient field in the triple u-metal shielded dewar, about 10^{-6} Tesla. After cooling the film, the temperature was raised above T_c and a known magnetic field applied by a superconducting coil below the sample. The sample was then cooled to 4.2K in the field, and then the field was removed. During this process the temperature pickup loop did not change by more than 0.01K, so the flux trapped in the loop should be minimal.

Figure 1 shows the change in flux in the pickup loop, $\Delta\phi$ in units of ϕ_0 , as a function of the temperature of the film at the pickup loop for a temperature gradient across the loop produced

by heating one side of the film after cooling in a 5 G field. This, of course, changes the temperature of the film at the loop, so comparisons must be made for a gradient with the same temperature as the temperature at the loop. This method of taking data was necessary since it is not possible to cool one end of the film, as would be required to establish a given temperature of the film at the center of the loop for a given gradient. Some data were taken with uniform heating, but in the case of Pb films the data were not quantitatively reproducible and are not shown. The data showed much more flux was moved by uniform heat than the same film temperature at the loop produced by a gradient. There appeared to be some strong dynamic, perhaps some instability, effect depending very strongly on the time dependence of the heating. This effect needs further study. No such effect was observed in Nb.

Figure 2 shows the same kind of data when the film is cooled in a 10 G field. The main thing to note is that the flux moved for a given gradient, or film temperature at the loop, does not scale linearly with the field, but increases considerably more rapidly. Figure 3 shows the data for a film cooled in a 22.5 G field, and again the flux which is moved increases more rapidly than the field.

Figure 4 shows the flux change at the loop for three different temperature gradients and temperatures at the loop as a function of H . The data are plotted as a function of the

temperature of the film at the loop, but the temperature difference across the loop can be read from the top scale of Figure 1. It should be pointed out that the sensitivity limit for the Pb data was about $10-15 \phi_0$, more than an order of magnitude smaller than that achieved later when the Nb data were taken.

NIOBIUM DATA

The Nb films used in this work were all 2000\AA thick films with nearly equiaxed grains of grain size about 280\AA , as determined by powder X-ray diffraction and STEM. This has been described in detail in previous reports to NCSC and NRL. The T_c 's of the films were in the range of 9.15 to 9.3K. Previous studies of pinning at high fields, as described in detail in previous final reports to NCSC and NRL, showed that the films were strong pinning materials in this regime, and that the grain boundaries were the primary pinning sites.

The data were taken in basically the same way as that for the Pb films described above, except the sensitivity (signal equal to noise) was $0.2-1 \phi_0$, making it possible to see effects at lower fields and smaller temperature gradients and changes. In the data for the Pb films, only irreversible flux motion was observed. At the higher sensitivity for the Nb films a small reversible signal was observed, but it was established that this was due to stray fields from the heaters. This reversible part is easily subtracted out as has been done for all the data

presented, giving only the irreversible motion of the flux trapped in the films. Unlike the Pb, the data are quantitatively reproducible for both temperature gradients and uniform heating.

The temperature gradients were established by heating one side of the film while the other side is kept at 4.2K, as measured by very small diode thermometers at each end of the film. Thus, assuming a uniform temperature distribution the change in temperature at the center of the loop is $(T_1 - T_2)/2$, and this is the temperature which should be compared to the equivalent temperature when both sides of the film are heated uniformly to simply raise the temperature of the entire film.

When films were cooled in the ambient field (about 0.01 G) no flux change was observed with either a gradient or uniform heating except very near T_c where fairly large fluctuations were observed. In all cases large fluctuations are observed near T_c , the magnitude correlating with the magnitude of the field in which the films were cooled.

Figure 5 shows the data for a film cooled in a 0.225 G field. No difference was observed in this low field regime between uniform heating and heating one side of the film to produce a temperature gradient. These data indicate that at this field the predominate mechanism for flux motion is the change in temperature of the film, not the temperature gradients.

Figure 6 shows the data for a film cooled in a 1.0 G field. For small temperature gradients or uniform heating ($\Delta T < .4K$) the

temperature of the film is the important factor in flux motion, while at higher temperatures and gradients, the gradient moves more flux.

Figure 7 shows the data for a film cooled in a 1.0 G field plotted as a function of the temperature difference across the pickup loop. The qualitative behavior is very similar to the plot as a function of the temperature at the center of the loop. Again, the predominate factor seems to be the change in temperature of the film at the loop, not the gradient, which moves flux at $\Delta T < 0.4K$.

The flux change in the pickup loop as a function of the temperature of the film for uniform heating and the temperature of the film at the center of the loop is shown in Figure 8 for a film cooled in a 5 G field. Considerably more flux is moved than for a 1.0 G field for a given temperature change or gradient, scaling roughly as the field, which is to be expected. Again, the hump at small changes in temperature is observed. The movement of flux by a uniform temperature change or a change produced at the loop by a gradient is the same in the low region, with the gradient producing a layer effect at larger changes.

The flux change at the loop as a function of the temperature difference across the pickup loop for a film cooled in a 5.0 G field is shown in Figure 9. Again, the qualitative behavior is the same as when the flux change is plotted as a function of the temperature at the center of the loop.

Figure 10 shows the same data as Figure 8 except the low temperature end has been expanded. The hump near 4.35K is reproducible and appears to be real. There are indications that there are at least two, and possibly three temperature regimes, where the driving mechanisms of the flux may be different. Figure 11 shows the flux change in the loop as a function of the temperature difference across the loop for the same region as Figure 10 to provide more detail at the lower gradients.

Figure 12 shows flux change through the loop for a film cooled in a 22.5 G field as a function of film temperature for uniform heating and temperature at the center of the loop when one side is heated. At this higher field the flux change is considerably larger, even at small changes, for a temperature gradient than a uniform change in temperature. Notice the hump seen for small temperature changes at lower fields is seen in the uniform heating data but not in the gradient data. The change in flux at the loop as a function of the temperature difference across the loop is shown in Figure 13.

Figure 14 shows the same data as Figure 12 except with the low temperature change region expanded. It can be seen in this figure that even with very small changes a gradient is much more effective in moving flux than an equivalent uniform temperature change, in contrast to the data at 5 G and below.

Figure 15 shows the same data as Figure 13 except the low gradient region has been expanded, showing more detail of the

hump at about 4mK across the loop.

Figures 16 and 17 show the flux change at the pickup loop due to given uniform temperature changes and temperature difference (from 4.2K) across the film as a function of the applied field. (Temperature difference across the loop is the temperature difference across the film divided by 17.)

DISCUSSION

As was expected all the flux changes at the loop due to flux motion was irreversible, that is cooling the film back to 4.2K did not change the flux in the loop after heating. This of course indicates that flux is swept out of the films.

The general trends of the data bring several interesting points to light. First of all, except at "high" fields and large gradients a change in the temperature produces as much flux motion as does a temperature gradient producing the same temperature at the loop. This effect is not understood, since the small amount of current theory only addresses thermal motion of fluxoids under the influence of temperature gradients. It may be that some kind of thermal equilibrium is established when the film is cooled through T_c to the final temperature of 4.2K, and when the temperature is raised this equilibrium is disturbed. This result certainly has implications in the operation of superconducting components and shields in SQUID magnetometer and gradiometer systems. There are also dynamic effects which could

not be shown in the quantitative data, since they depend on the rate of heating and are not totally reproducible. Undoubtedly some of these are due to thermal time constants and other characteristics in the system, but we are convinced some of the effects are due to the film. In further studies to characterize the noise in SQUID based systems, these effects may be found to be important. We are currently considering methods of studying these effects quantitatively.

As can be seen from Figures 4, 16, and 17 for a given field and a given temperature gradient, considerably more flux is moved in the Pb films than in the Nb films. This correlates with the previous high field data showing these Nb films to be strong pinning, while the Pb is expected to be weak pinning. Obviously, at least for film materials, Nb makes an appreciably better magnetic shield material than Pb if one wishes to minimize flux motion with temperature changes and temperature gradients. It will be extremely interesting to determine if these results carry over into bulk materials.

Another interesting aspect of the data is the "knee" or "bump" in all the Nb data at small temperature gradients or temperature changes. The origin of this bump is not currently understood. Also, the behavior is quite different in the small temperature change or gradient region than in the high, and the low and high field regions. Further work is clearly required to understand this behavior and apply the results to superconducting

components and shields.

FIGURES AND CAPTIONS

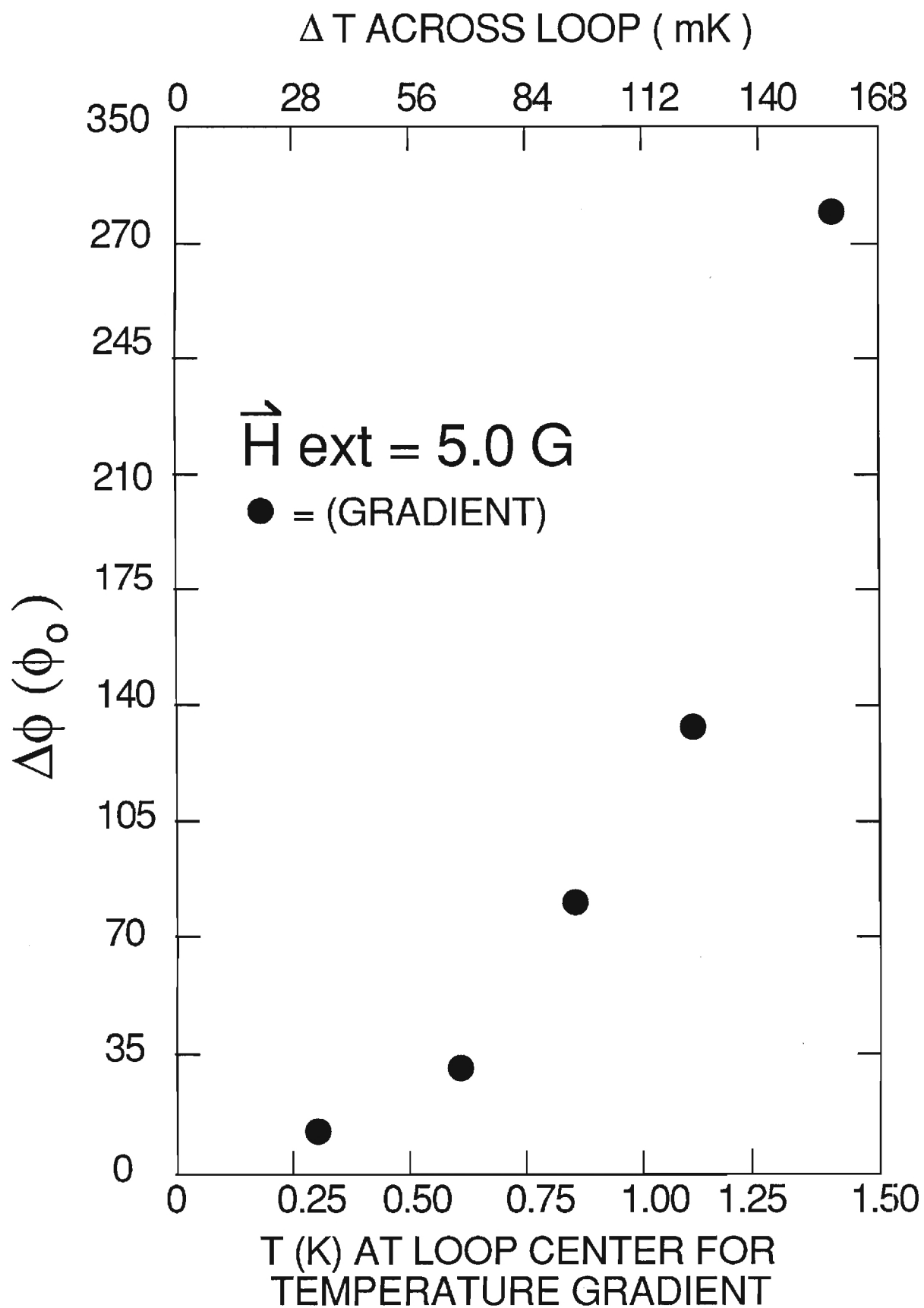


FIGURE 1

1. Flux change, $\Delta\phi_0$ in units of ϕ_0 , in the pickup loop for a 2000Å Pb film cooled in a 5.0 G field as a function of temperature and temperature gradient. The top scale is the temperature difference across the loop and the lower one is the change in temperature at the center of the loop (from 4.2K) when a temperature gradient is applied by heating only one side of the film. To get the temperature at the loop, add 4.2K to the lower axis.

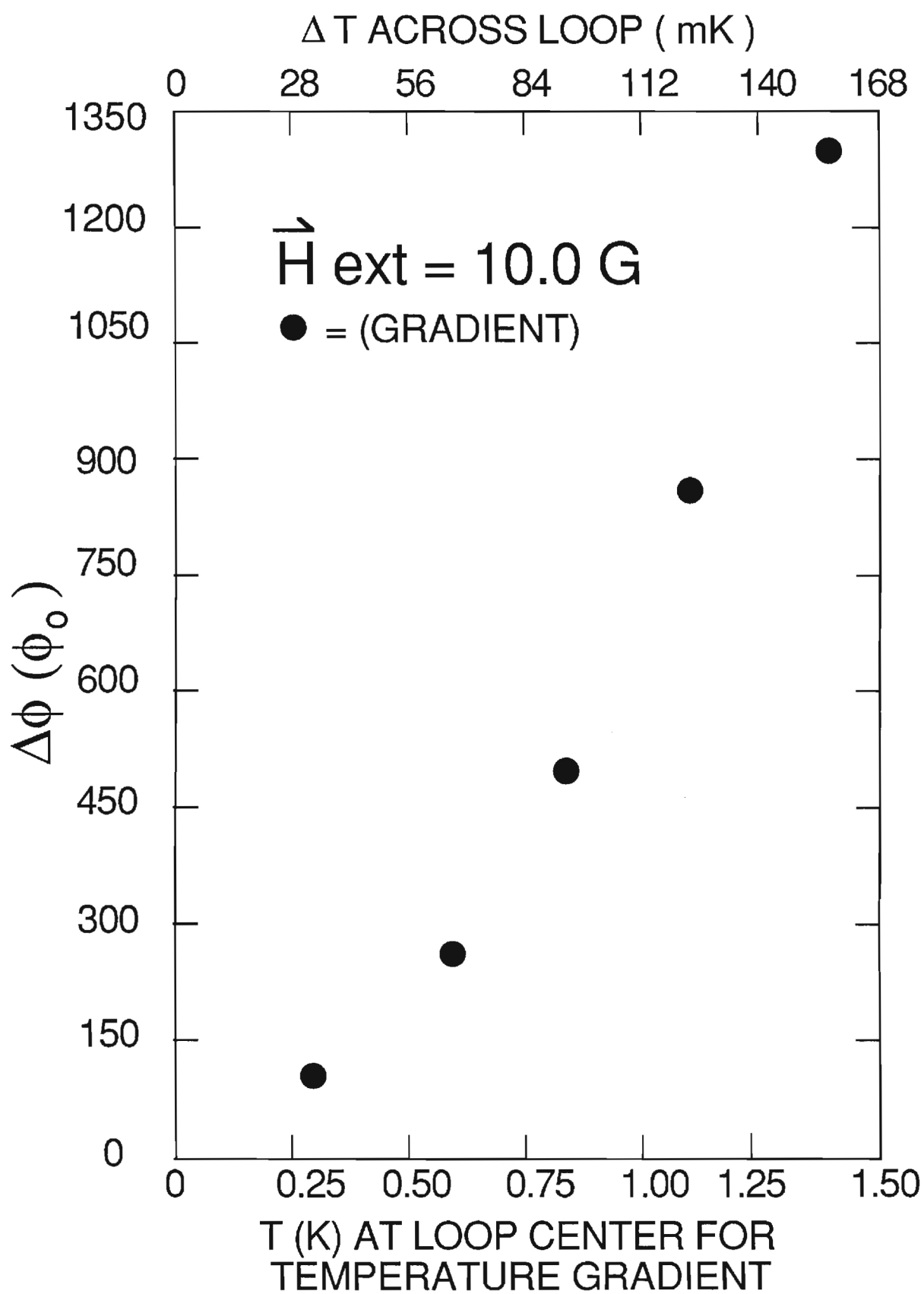


FIGURE 2

2. Flux change, $\Delta\phi_0$ in units of ϕ_0 , in the pickup loop for a 2000\AA Pb film cooled in a 10.0 G field as a function of temperature and temperature gradient. The top scale is the temperature difference across the loop and the lower one is the change in temperature at the center of the loop (from 4.2K) when a temperature gradient is applied by heating only one side of the film. To get the temperature at the loop, add 4.2K to the lower axis.

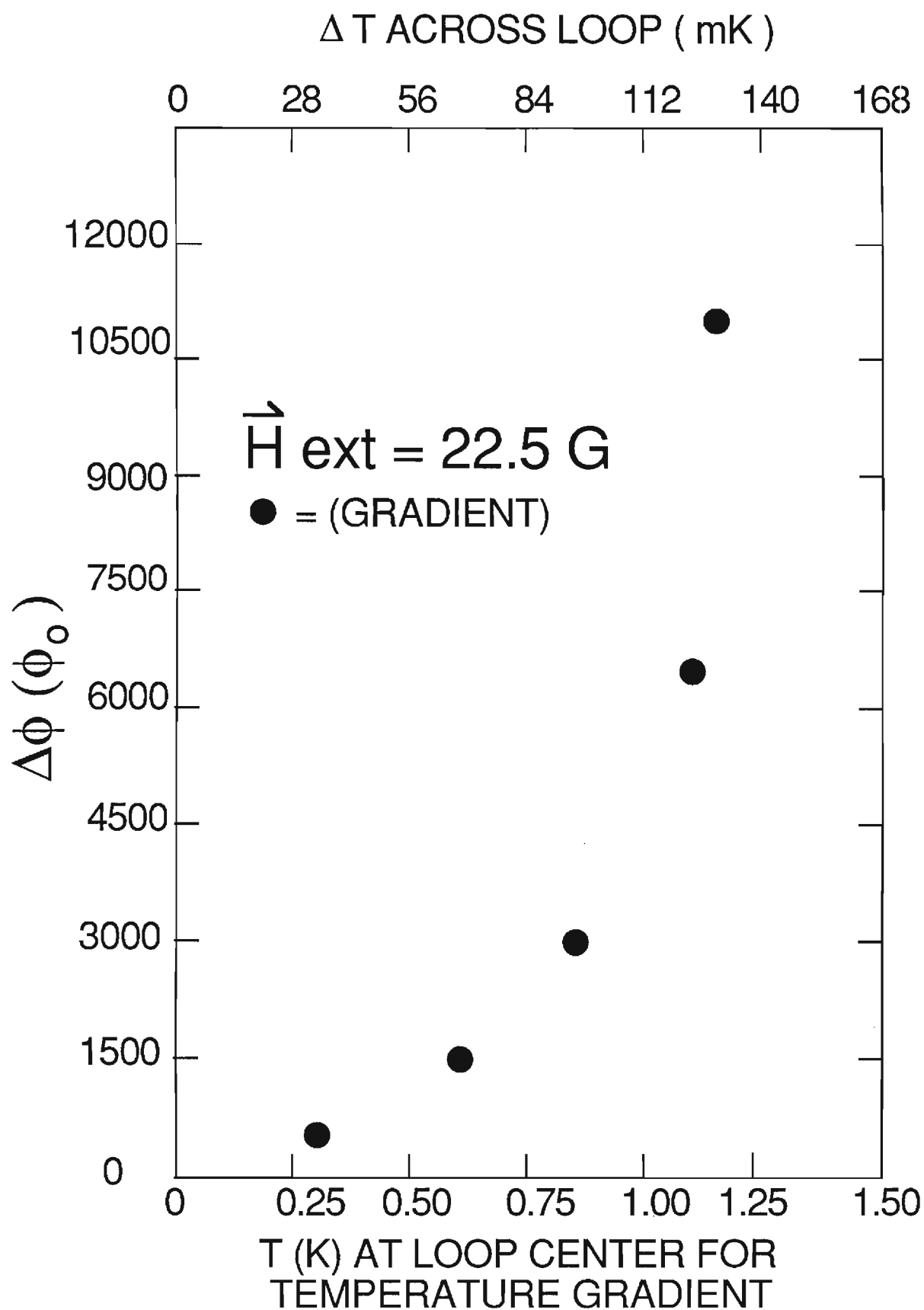


FIGURE 3

3. Flux change, $\Delta\phi_0$ in units of ϕ_0 , in the pickup loop for a 2000Å Pb film cooled in a 22.5 G field as a function of temperature and temperature gradient. The top scale is the temperature difference across the loop and the lower one is the change in temperature at the center of the loop (from 4.2K) when a temperature gradient is applied by heating only one side of the film. To get the temperature at the loop, add 4.2K to the lower axis.

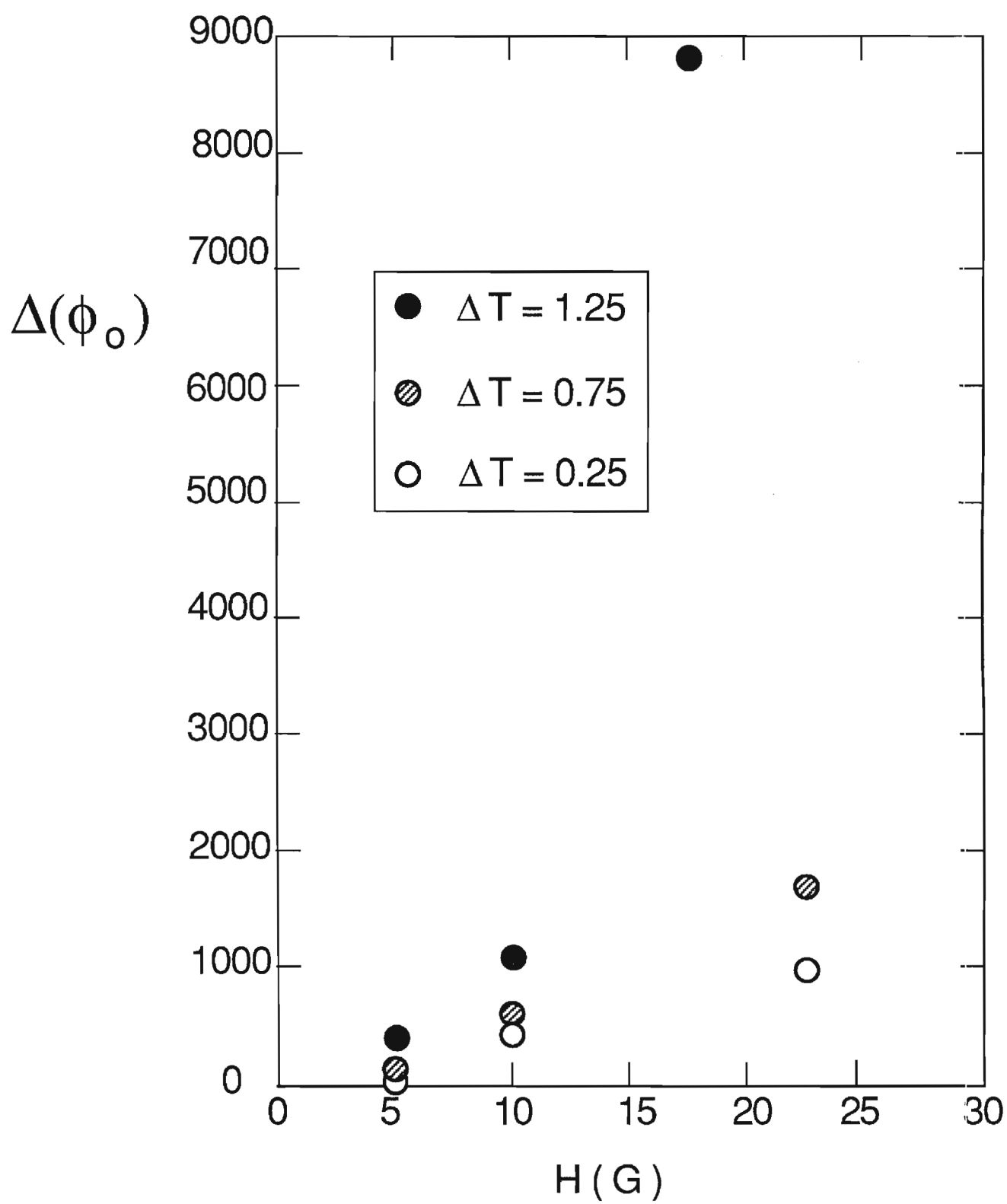


FIGURE 4

4. Flux change in the pickup loop as a function of the field in which the film was cooled at three different film temperatures at the loop with a gradient applied. The temperature at the loop is $\Delta T + 4.2\text{K}$. The temperature difference across the loop may be obtained by using the upper and lower ordinates of Figure 1.

T (K) OF FILM FOR UNIFORM HEAT
OR AT CENTER OF LOOP FOR T GRADIENT

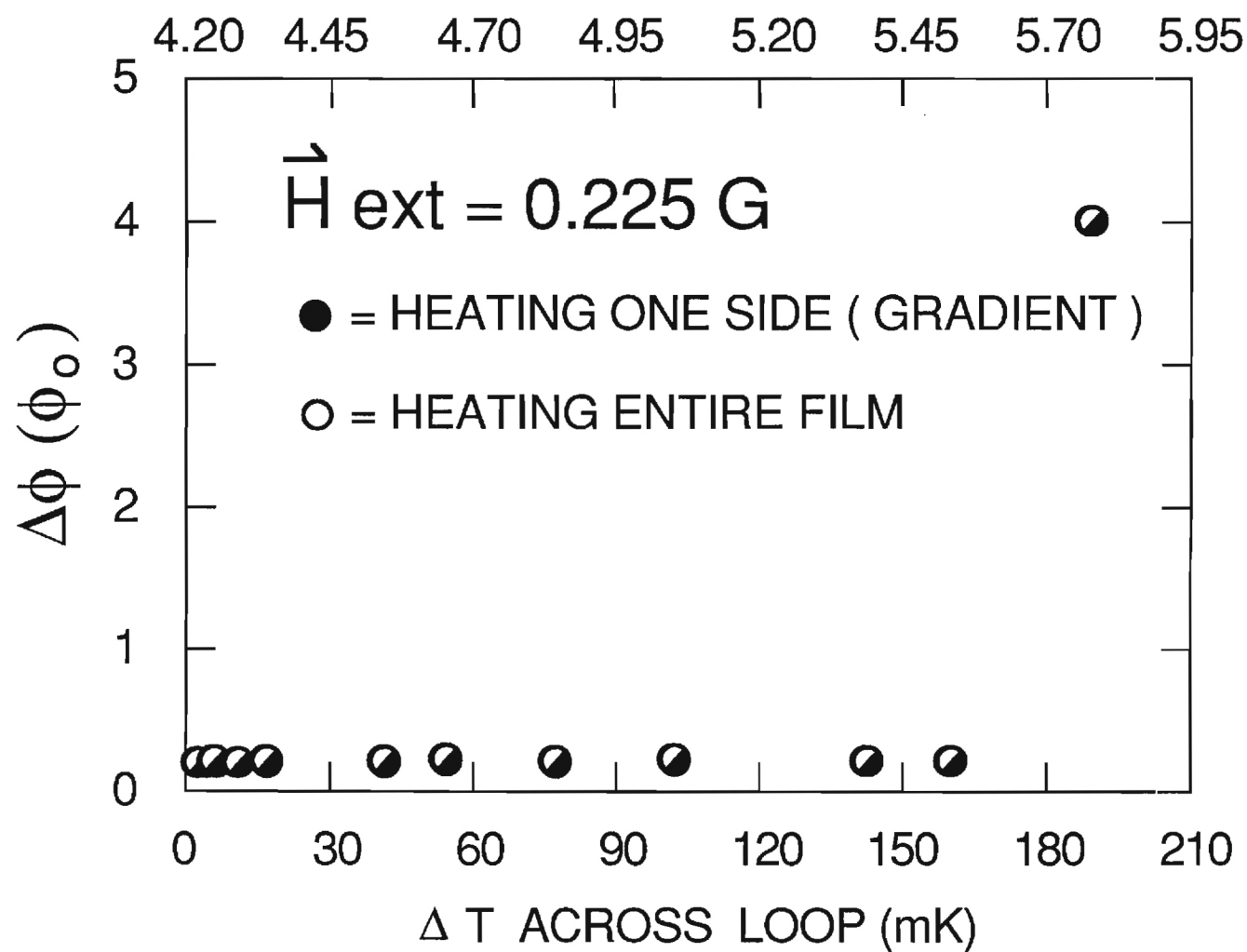


FIGURE 5

5. Flux change in loop for 2000\AA Nb film cooled in 0.225 G field. The top temperature scale shows the temperature of the film when uniformly heated and the temperature at the center of the loop when one side is heated. The lower temperature scale shows the temperature gradient across the loop.

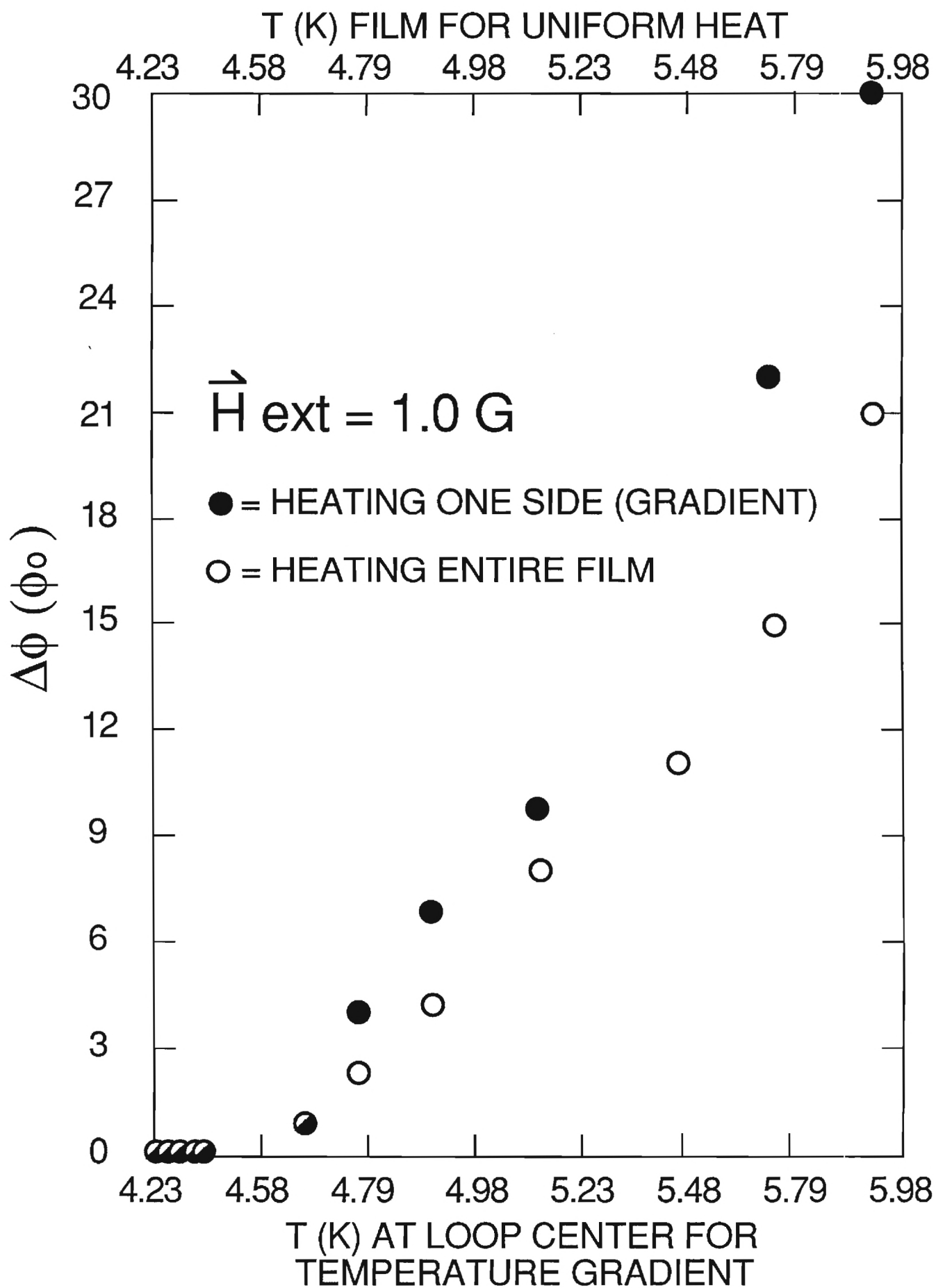


FIGURE 6

6. Flux change through pickup loop for a Nb film cooled in a 1.0 G field as a function of temperature of the film (uniform heating), or the temperature at the center of the loop for a temperature gradient.

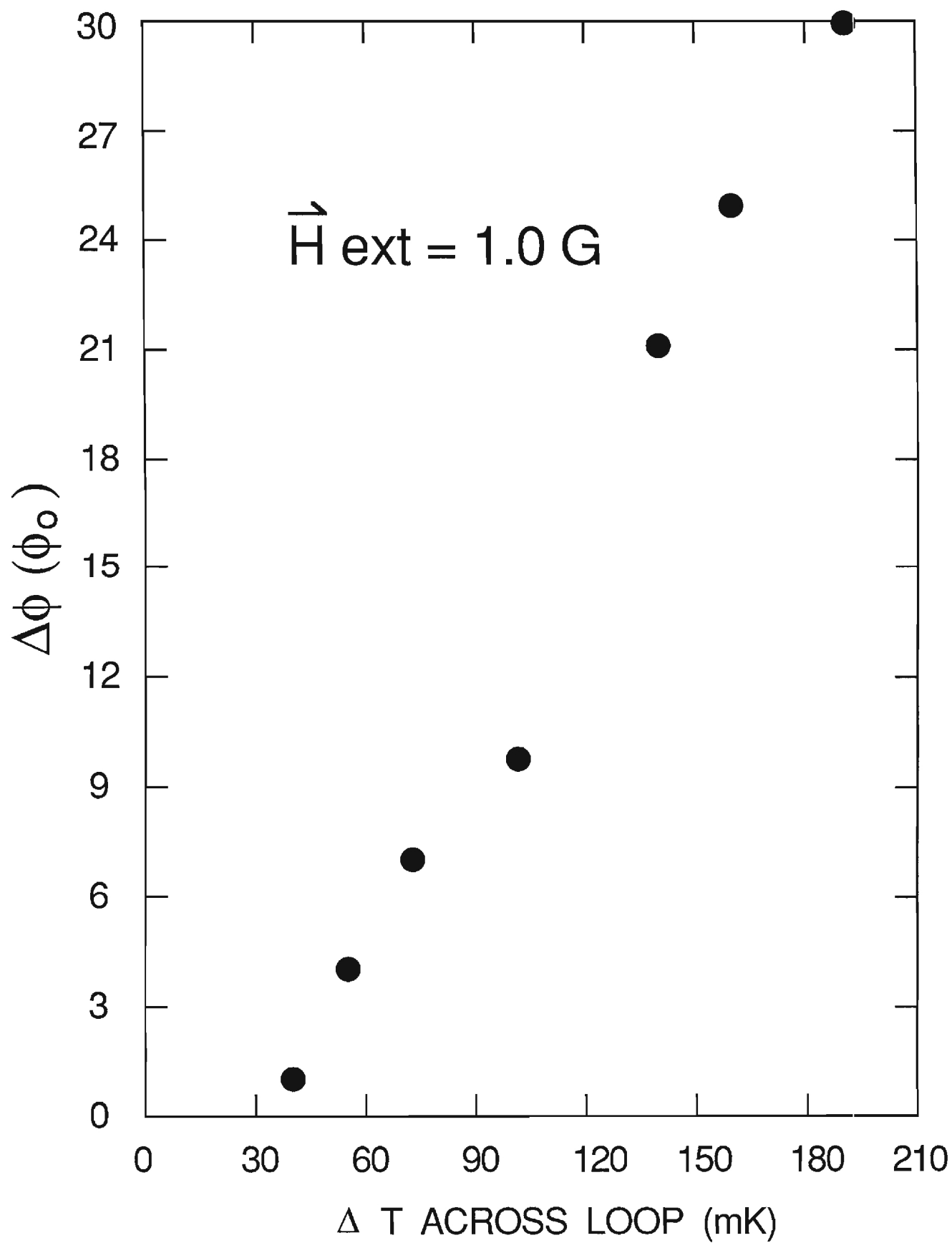


FIGURE 7

7. Flux change through pickup loop for a film cooled in a 1.0 G field as a function of the temperature difference across the pickup loop produced by heating one side of the film only.

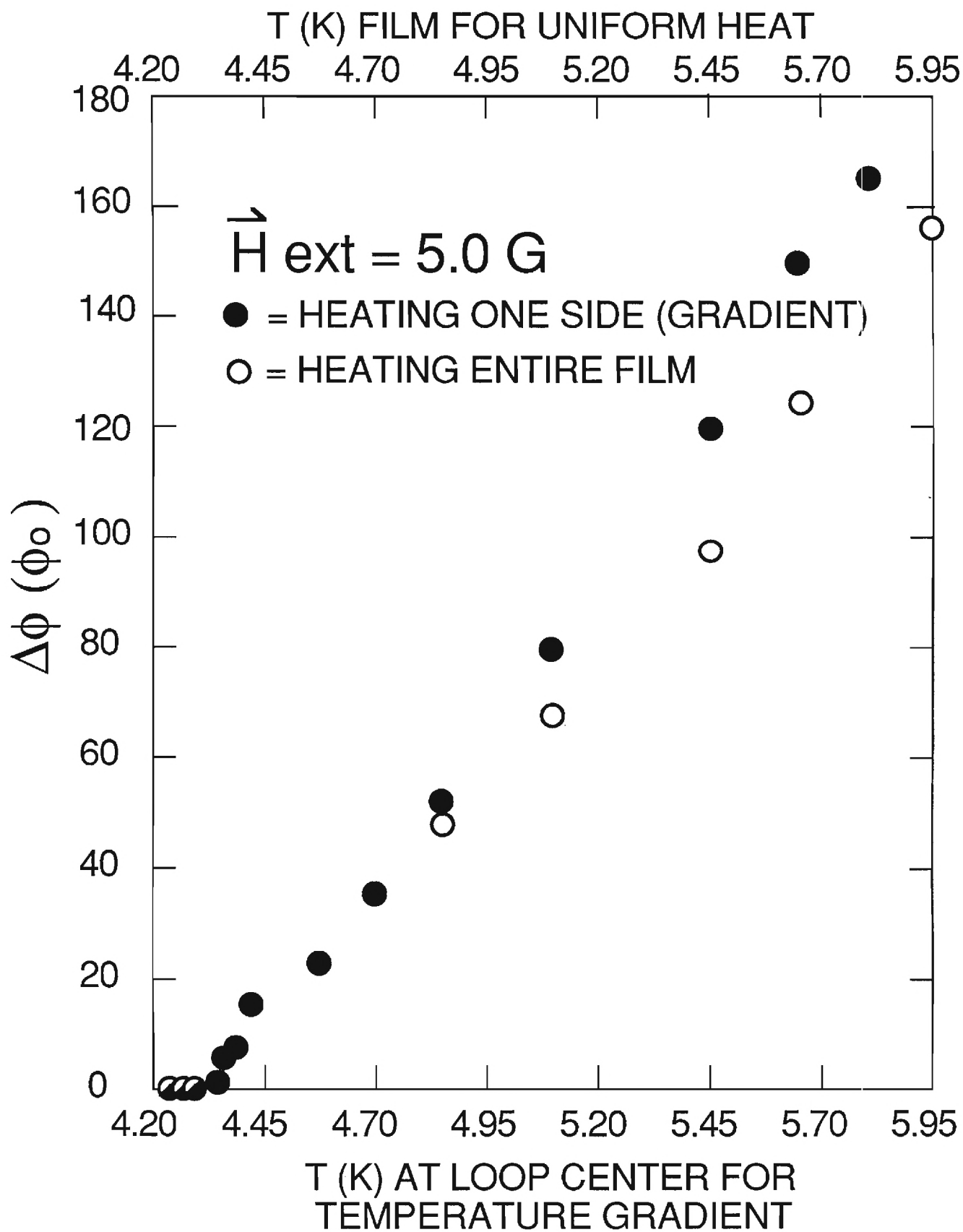


FIGURE 8

8. The change in flux in the pickup loop as a function of film temperature (uniform heating) and the temperature at the center of the loop when one side is heated producing a gradient for a film cooled in a 5.0 G field.

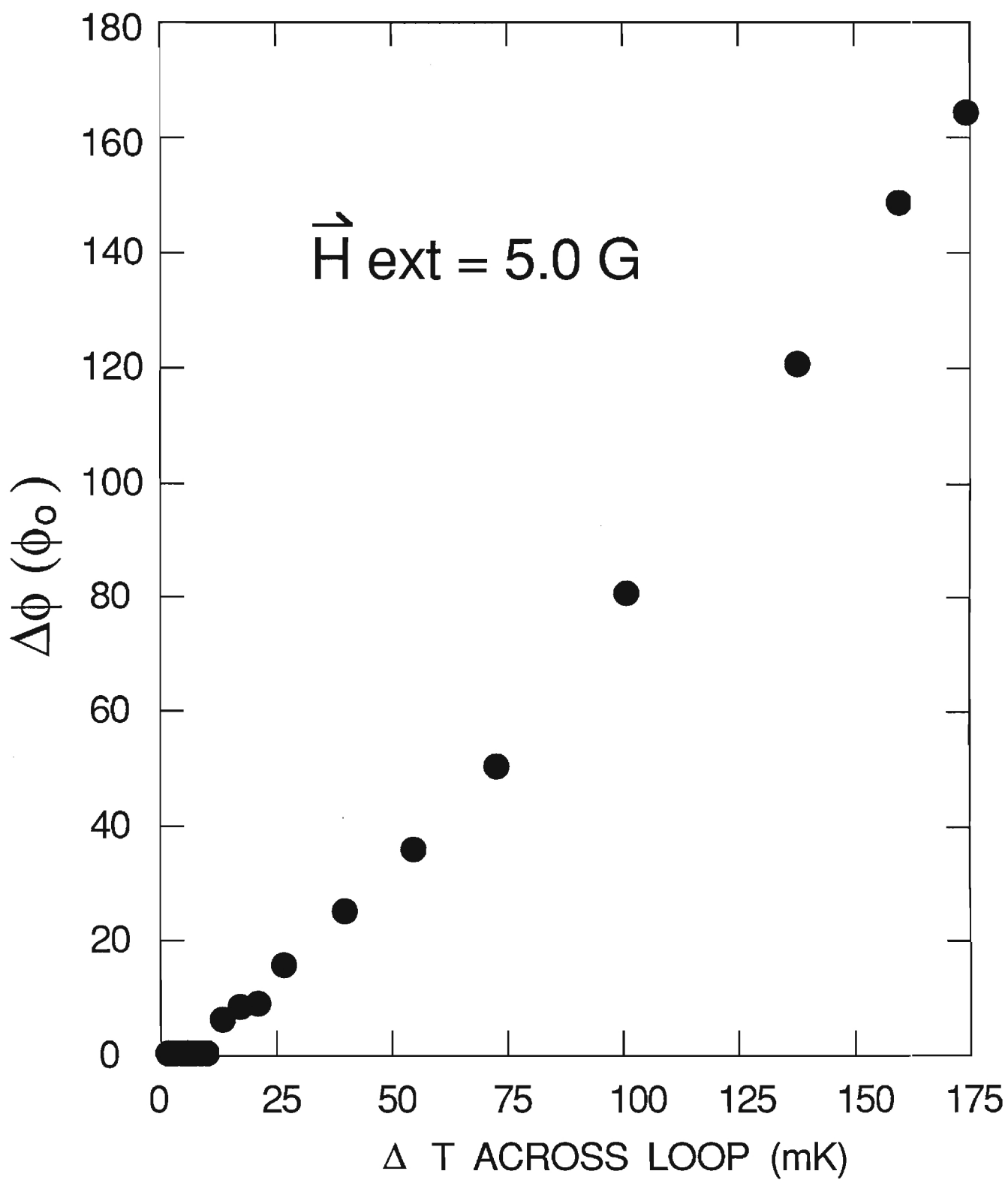


FIGURE 9

9. The change in flux in the pickup loop as a function of the temperature difference across the pickup loop for a film cooled in a 5.0 G field.

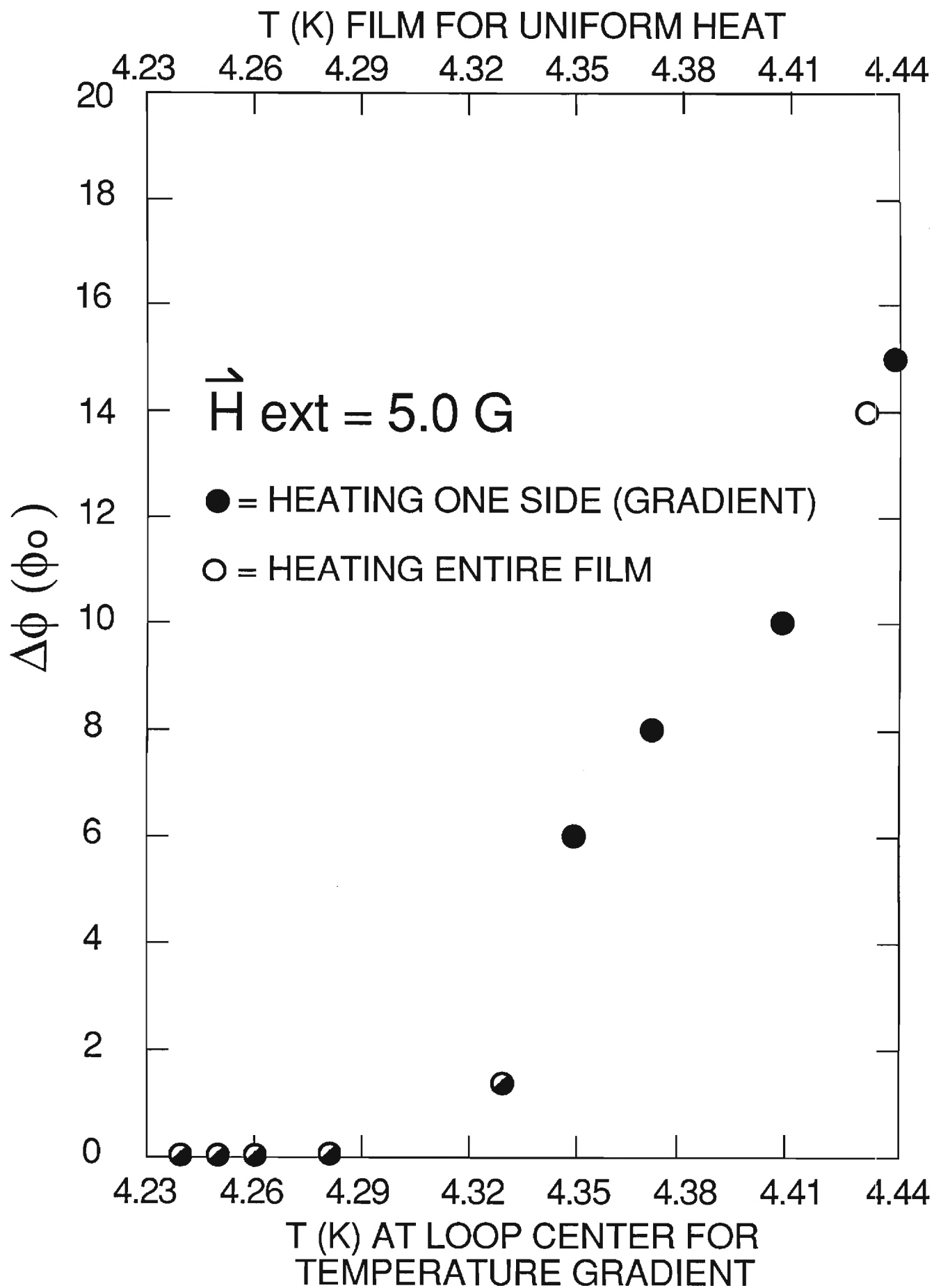


FIGURE 10

10. Same data as seen in Figure 8, but with the low temperature region expanded to see the detail for small temperature changes more clearly.

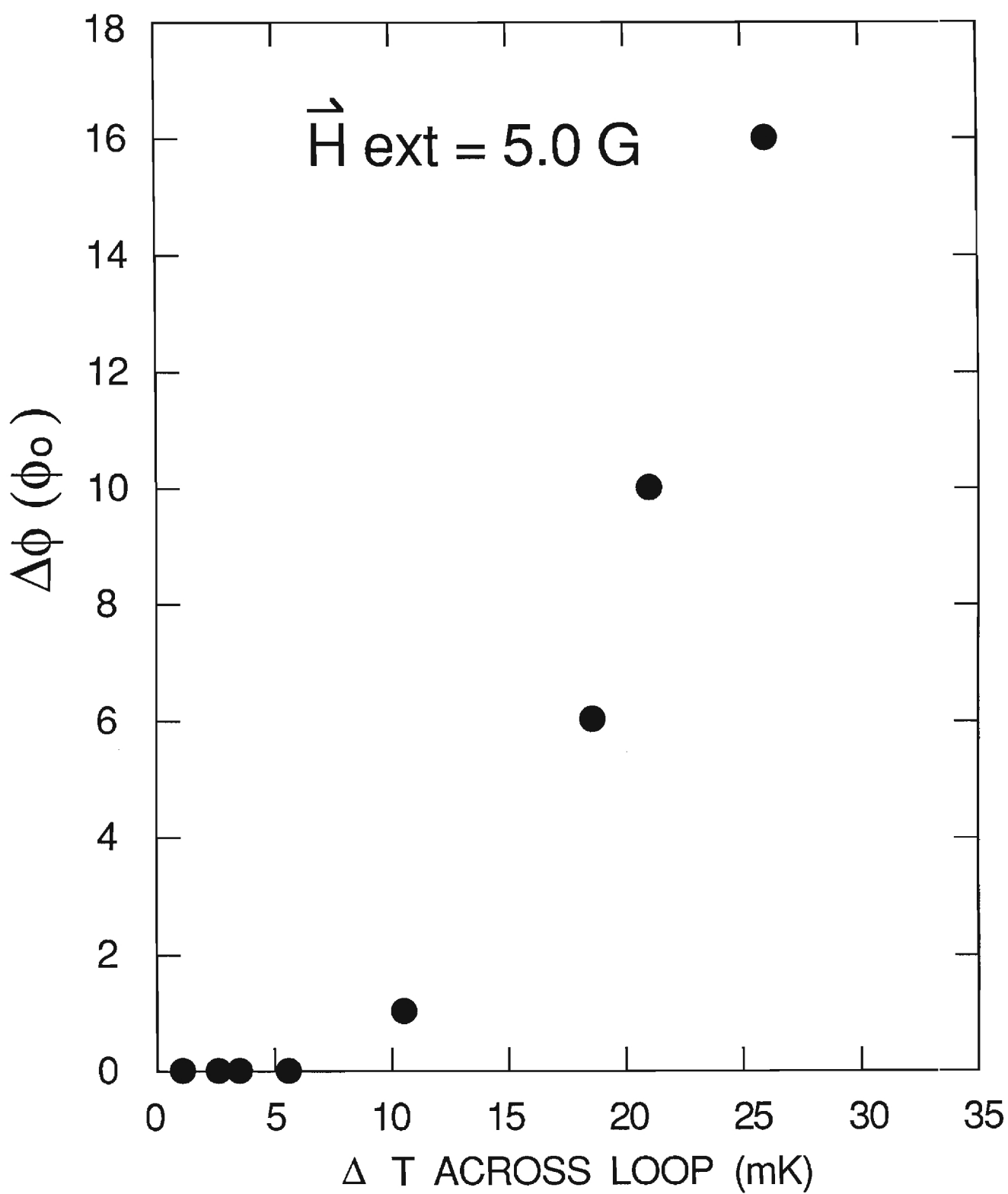


FIGURE 11

11. This shows the same data as seen in Figure 9, but the low temperature region is expanded to correspond to Figure 8.

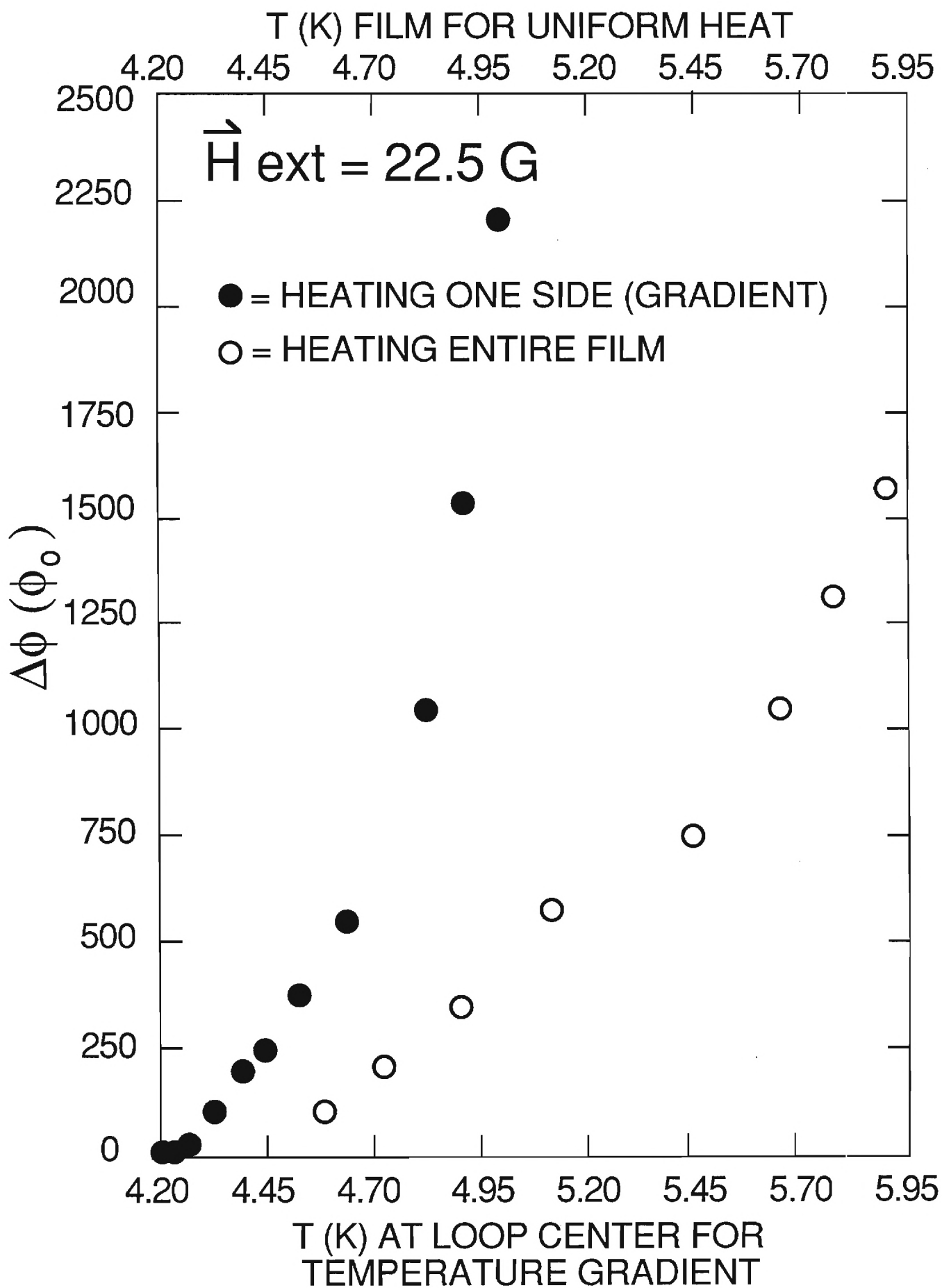


FIGURE 12

12. Flux change, for film cooled in 22.4 G field, through the pickup loop as a function of temperature for uniform heating, and temperature at the center of the loop for heating one side of the film.

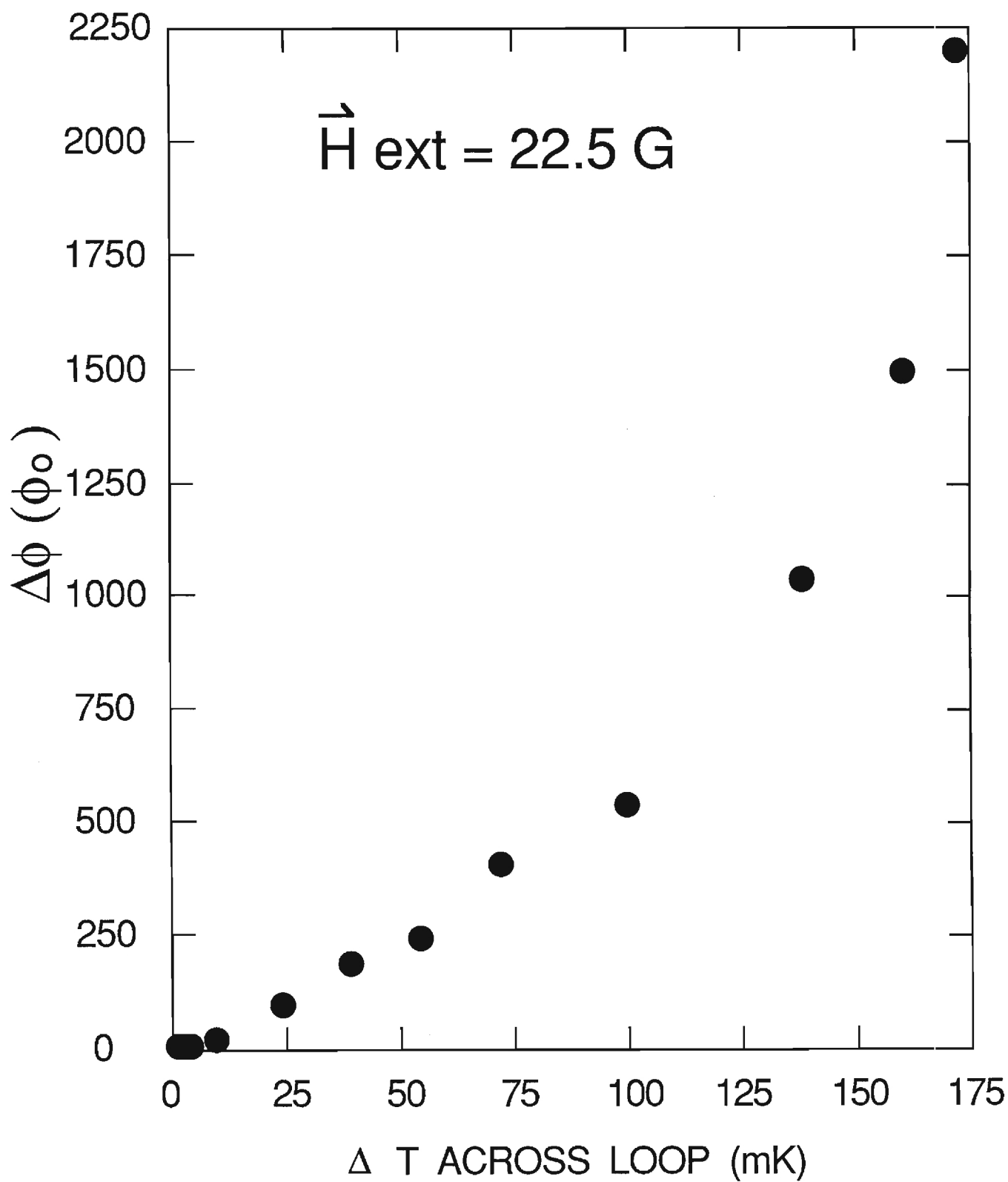


FIGURE 13

13. Flux change in the pickup loop as a function of temperature difference across the loop for a film cooled in a 22.5 G field.

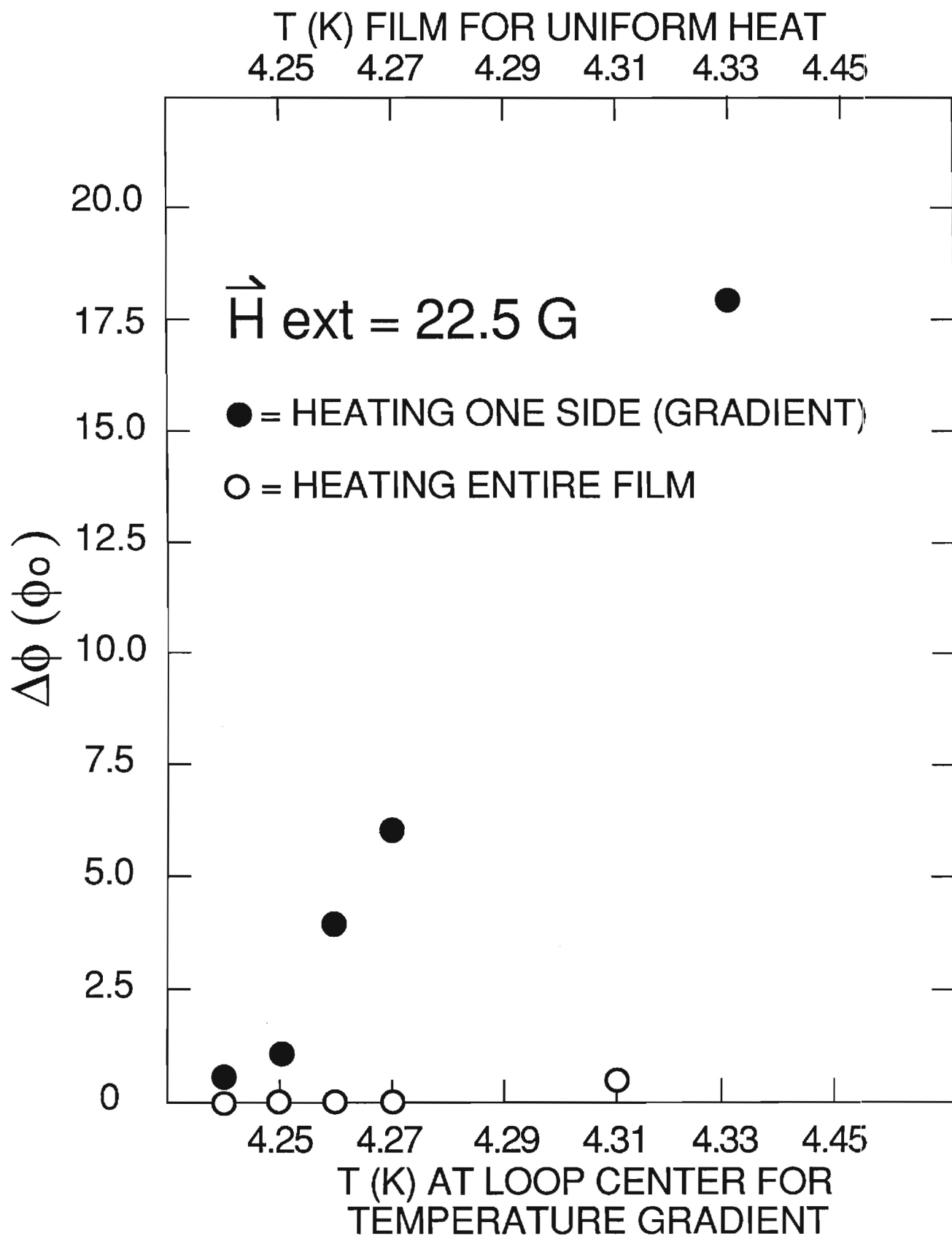


FIGURE 14

14. Same data as Figure 12, except the low temperature region is expanded to show the details in this region.

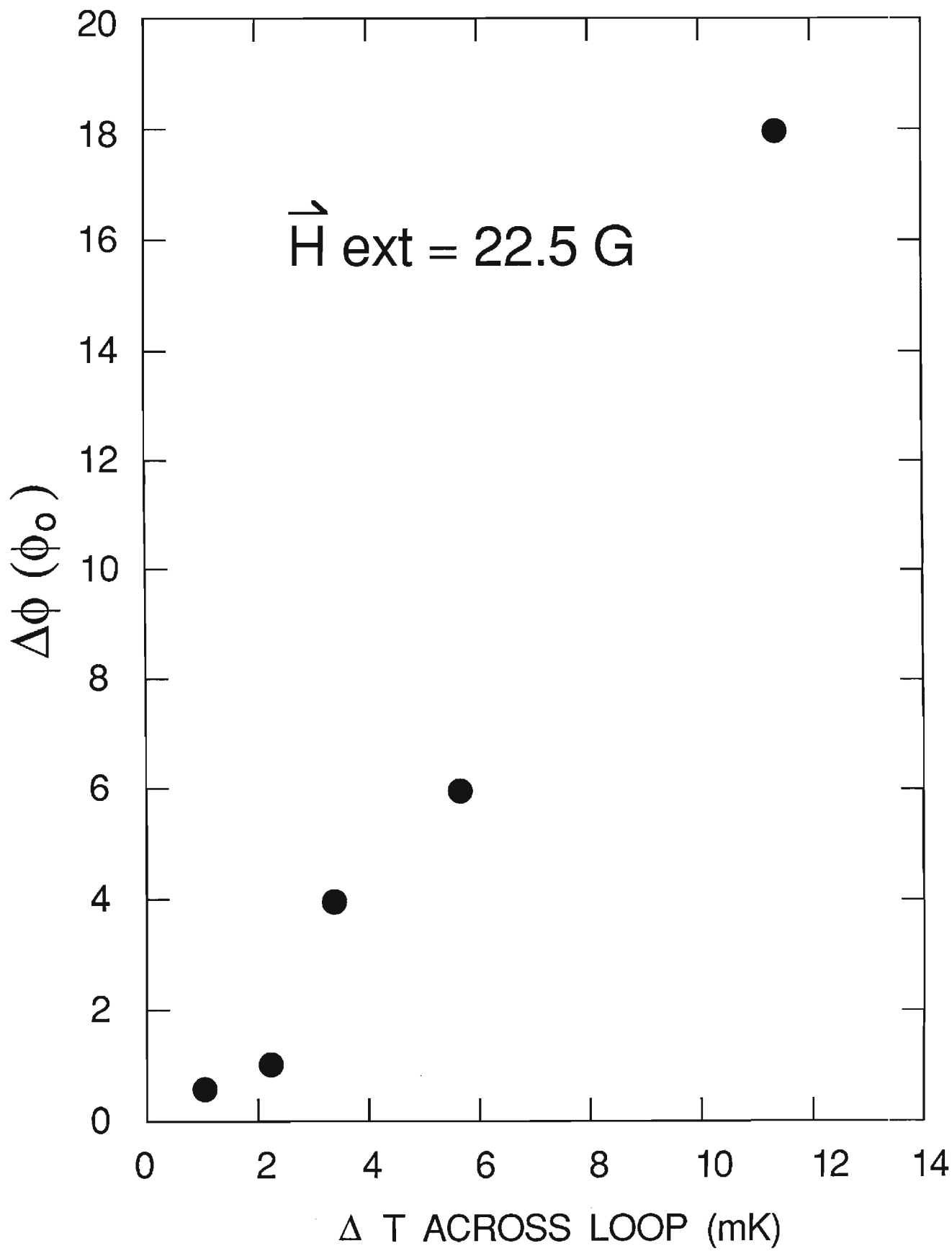


FIGURE 15

15. The data is the same as Figure 13, except the small temperature gradient region is expanded.

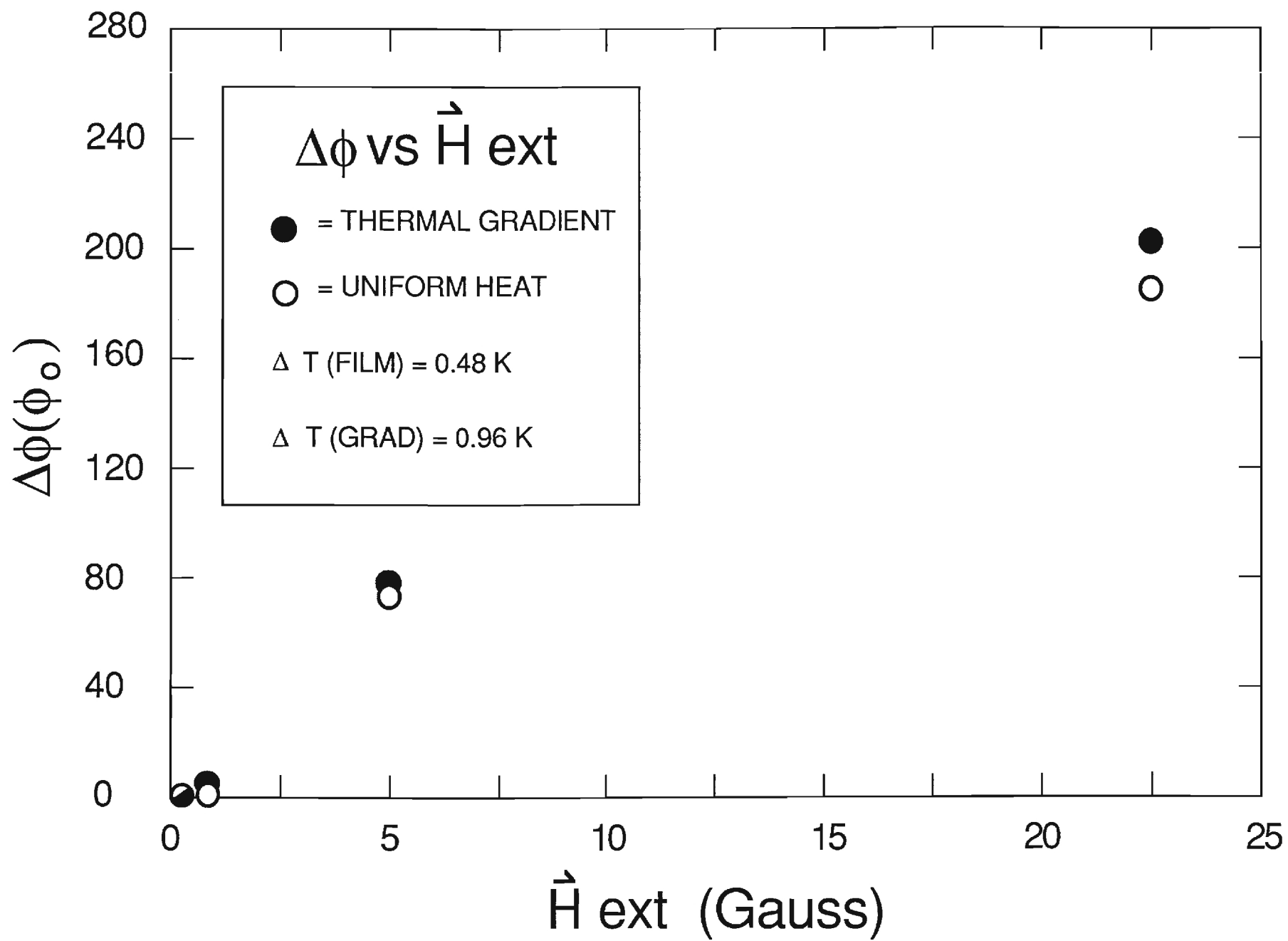


FIGURE 16

16. Flux change at the pickup loop as a function of field in which the film was cooled to 4.2K for temperature at the loop of 0.4K, and for 0.96K temperature difference for the temperature gradient data. The temperature difference across the loop for the gradient data is 5.6 mK.

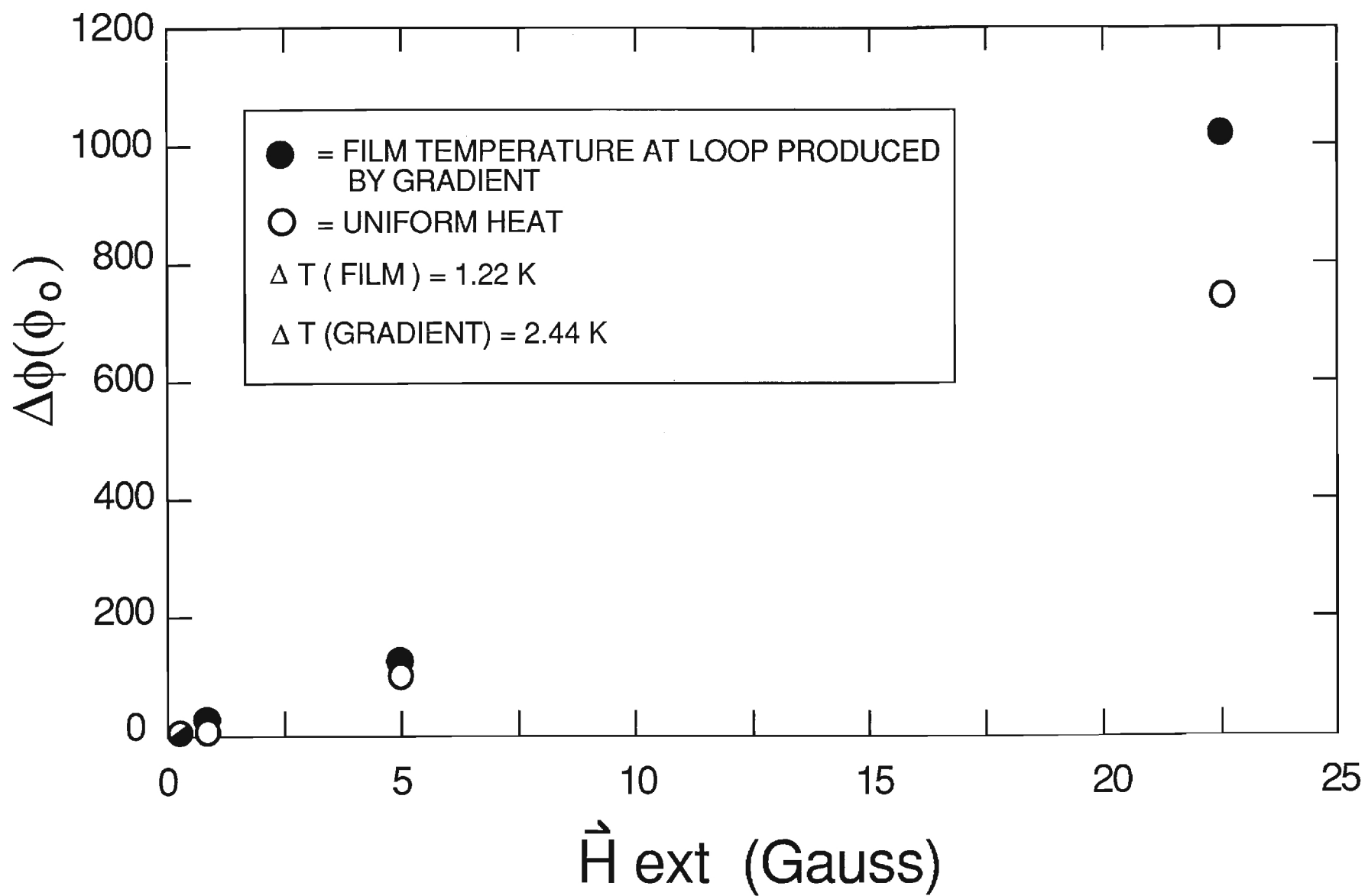


FIGURE 17

17. Flux change at pickup loop as a function field in which the film was cooled to 4.2K for temperature at the loop of 1.22K for uniform heating or gradient. The temperature difference across the loop was 14 mK.